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(पहला पुनरीक्षण)

Indian Standard

METHODS OF CHEMICAL ANALYSIS OF ZINC AND ZINC BASE ALLOYS FOR DIE CASTINGS

PART 4 DETERMINATION OF CHROMIUM BY SPECTROPHOTOMETRIC METHOD

(First Revision)

UDC 669·5 : 543·42 [546·76] : 621·74·043

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

February 1993 **Price Group 1**

Methods of Chemical Analysis of Non-Ferrous Metals Sectional Committee, MTD 28

FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Methods of Chemical Analysis of Non-Ferrous Metals Sectional Committee had been approved by the Metallurgical Engineering Division Council.

IS 2600 was first published in 1964. It covers the methods for determination of copper, tin, iron, aluminium, magnesium, cadmium and lead in high purity zinc and zinc base alloys for die castings. The Committee decided to revise this standard into parts, which on publication would supersede the determination of that element given in IS 2600 : 1964. During this revision the method for determination of nickel, thallium, indium and chromium in zinc and zinc base alloy castings has been added.

This standard (Part 4) covers the method for determination of chromium by spectrophotometric method. The other parts of the series are as follows:

IS 2600	Methods of chemical analysis of zinc and zinc base alloys for die castings:
Part 1	Determination of copper, iron, nickel, tin and thallium by spectrophotometric method
Part 2	Determination of copper, iron, lead and cadmium by atomic absorption spectrometric method
Part 3	Determination of indium by atomic absorption spectrometric method
Part 5	Determination of aluminium and magnesium by atomic absorption spectrometric method

In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

Indian Standard

METHODS OF CHEMICAL ANALYSIS OF ZINC AND ZINC BASE ALLOYS FOR DIE CASTINGS

PART 4 DETERMINATION OF CHROMIUM BY SPECTROPHOTOMETRIC METHOD

(First Revision)

1 SCOPE

This standard (Part 4) describes the method for determination of chromium up to 0·02 percent.

2 REFERENCES

The following Indian Standards are necessary adjuncts to this standard:

IS No.	*Title*
264 : 1976	Nitric acid (*second revision*)
1817 : 1961	Methods of sampling non-ferrous metals for chemical analysis
1070 : 1992	Reagent grade water (*third revision*)

3 SAMPLING

Sample shall be drawn and prepared in accordance with the procedure laid down in IS 1817 : 1961.

4 QUALITY OF REAGENTS

Unless specified otherwise, analytical grade reagents and distilled water (*see* IS 1070 : 1992) shall be employed in the test.

5 DETERMINATION OF CHROMIUM BY SPECTROPHOTOMETRIC METHOD

5.1 Outline of the Method

The sample is dissolved in dilute sulphuric acid and a few drops of nitric acid and allowed to fume. It is then diluted with distilled water and boiled to dissolve salts. Chromium is oxidized with dilute potassium permanganate solution, excess of which is removed with sodium azide. An aliquot of this solution is treated with diphenyl carbazide and the absorbance measured at 540 nm.

5.2 Reagents

5.2.1 *Dilute Sulphuric Acid*, 1 : 4 (*v*/*v*)

5.2.2 *Nitric Acid*, rd = 1·42 (Conforming to IS 264 : 1976).

5.2.3 *Potassium Permanganate Solution*, 0·1 N

5.2.4 *Sodium Azide Solution*, 0·5 percent.

5.2.5 *Diphenyl Carbazide Solution*, 0·25 percent in acetone.

5.2.6 *Standard Chromium Solution* (1 ml = 0·000 1 g Cr)

Dissolve 0·025 g of pure chromium in 10 ml of dilute sulphuric acid (1 : 4). Add a few drops of nitric acid after completion of reaction. Fume out the nitric acid. Cool and make up to 250 ml.

5.2.6.1 *Standard chromium solution* (1 ml = 10 μg Cr)

Dissolve 10 ml of standard chromium solution (**5.2.6**) in 100 ml of water.

5.2.6.2 *Standard chromium solution* (1 ml = 1 μg Cr)

Dissolve 10 ml of standard chromium solution (**5.2.6.1**) in 100 ml of water.

5.3 Procedure

5.3.1 Weigh accurately 2·0 g of the sample. Dissolve in dilute sulphuric acid (1 : 4). Add a few drops of nitric acid. Take to fumes. Dilute with water and boil. Add potassium permanganate solution (0·1 N) drop by drop till a pink colour persists. Continue to boil for 10 minutes. Make sure that the pink colour persists during boiling.

To the boiling solution add sodium azide solution (0·5 percent) drop by drop till the permanganate colour disappears completely. Avoid excess of the azide. Cool and make up to 100 ml. Filter, if suspended particles are present. Take 20 ml of the clear made up solution. Add 1 to 2 ml of diphenyl carbazide solution and make up to 25 ml. Measure the absorbance at 540 nm.

5.3.2 Blank

Carry out a blank determination using same amount of reagents and following the procedure as specified in **5.3.1** but without the sample.

5.3.3 Calibration

Take 0, 1, 2, 3 and 4 ml of the standard chromium solution (**5.2.6.2**) and proceed as described in **5.3.1.** Note the absorbance of each solution and draw a calibration curve with milligrams of chromium and absorbance.

5.4 Calculation

Convert the spectrophotometric reading of the sample into milligrams of chromium from the calibration curve and calculate the percentage of chromium as follows:

$$\text{Chromium, percent by mass} = \frac{M}{m} \times 100$$

where

M = mass, in mg, of chromium found; and

m = mass, in mg, of the sample taken.

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